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X-RAY METHOD FOR DETERMINING FILM THICKNESS BY MEANS OF SUPERIMPOSITION

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In 1940 we suggested an X-ray method for determining film thickness (1, 2) which was subsequently used at the Khar'kov Electromechanical Plant. In this method the film thickness is determined by formula after taking an X-ray photograph by the slide procedure.

We also examined two-layer crystalline films and the case of an amorphous film whose absorption coefficient is known (1). Subsequently (3), we used the X-ray method to determine the concentration of the crystalline phases and the thickness of a nonuniform surface layer on a crystalline base containing or not containing one of the phases of this layer. In particular, we discussed the case where the base, carbon steel, consists of the alpha phase and cementite, and the thin surface layer is composed of the residual austenite and its transformation products.

In order to use the above-mentioned formula, it is necessary to know the minute structure of the substances forming the film and the base. Moreover, the computations are very cumbersome, especially when complex structures are present, oxides of metals, carbides, etc. The application of the X-ray method is limited by effects which distort the normal intensity of the Debye lines (1). Such effects are local amplification or weakening of the interference maximums due to the texture or large-grain size of the materials and the relative changes in the blackening of the Debye lines at various angles of reflection, due to increased dispersion, a strained condition of the crystalline phases, or unevenness of the solid solution.

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A method is suggested below to widen the field of application of the X-ray method for determining the thickness of films and for simplifying the calculation formulas. This method is applicable if the film or base consists of a complex crystalline phase whose structure has not been determined; if the film or base contains a uniform mixture of several phases, one of which may be amorphous; and also if the above-mentioned effects exist in the film or base.

As in the previous papers (1, 3), we have deduced the formulas for the slide method. This method gives the formulas the simplest possible form. Moreover, in most cases the slide shape is the most convenient on which to deposit films. If another X-ray method is employed, it is necessary to introduce corresponding corrections into the formulas.

Differential Method

This method utilizes the effect of variations in the ratio of the intensities of the X-ray reflections from the film and the base during changes in the wave length of the primary radiation (3, 4). This effect depends on the thickness of the film and on the corresponding absorption coefficients of the X rays.

The use of two radiations enables two systems of Debye lines to be identified on the roentgenogram of the specimen under examination, and it is possible to tell which of them corresponds to the film and which to the base. Moreover, if the chemical compositions of the film and base are known approximately, and if it is possible to assume that certain conditions remain the same in passing over from one radiation to another, it is not necessary to know their structure. However, the application of this method is limited by the necessity for very exact measurement of the intensity of the reflections, since it varies very slightly with the wave length of the X rays.

Method of Standard Mixtures

The difficulties associated with the calculation of the structural factor and other quantities entering into the formulas derived by us can be overcome by taking a roentgenogram of a mixture whose structural components are the same substances as those of the film and base.

The drawback of this method is that the preparation of the mixtures requires careful mixing of both phases. This is not always possible. Moreover, the method does not make allowances for texture, which is not infrequently observed in various films and bases.

Superimposition Method

This method also is based on experimental measurements of the intensities of the Debye lines for the substances in the film and base. The method consists of taking both substances on the same roentgenogram which results in a picture resembling those obtained from mixtures. During exposure, the two substances are alternated through definite time intervals whose ratio determines the ratio of the integral intensities of the pair of Debye lines being compared.

In order to obviate the influence of the sensitivity of the X-ray film with time and the influence of fluctuations during the operation of the X-ray tube, it is necessary that these time intervals be sufficiently small with respect to the total exposure time of the roentgenogram. For example, when investigating cylindrical specimens (5) it is possible to use Sekito's method which is employed in phase analysis (6). It is also easy, when using the slide method, to make suitable arrangements for periodically interchanging both substances while exposing them for any small time intervals desired.

One of these substances can be a thick film with the same composition, structure, and peculiarities, e.g., texture, dispersion of crystals, etc., as the film whose thickness is to be determined. The second substance can be the specimen being investigated, but without the film. The ratio of the integral intensities of the Debye lines on a roentgenogram taken by the method of superimposition can then be determined by formula.

Thus the superimposition method, which has all the advantages of the method of standard mixtures, automatically takes into account the structural condition of the base and the film -- texture, mosaic, coarse-grain, dispersion, strain, disequilibrium of solid solution, etc. This is important because all the above effects, especially texture, often occur when the film is deposited in widely different ways, e.g., by electrolysis (6, 7) condensation of vapors in a vacuum (8, 9), oxidation of metals (10), etc., and can be properly allowed for. In some cases the influence of texture can even be used to increase the sensitivity of the roentgenogram method of determining the thickness of coverings.

The method can also be used for quantitative phase analysis, where the unknown quantities will be the relative concentrations of standard mixtures in the alloy being investigated.

Assessing Accuracy and Sensitivity of X-Ray Method of Determining Film Thickness

The main error which determines the accuracy of the measurements of film thickness is the error in measuring the intensity of the Debye lines.

Application of Superimposition Method to Measurement of Effective Thickness of an Oxide Layer on Steel

We are using the superimposition method in various cases of phase analysis and determination of the thickness of crystalline films. A rapid X-ray method for mass control of the effective thickness of the oxide layer on oxidized steels is described below. For this purpose we used the high speed Bolinovskiy camera, developed by us at Khar'kov Electromechanical Plant in 1939, which uses the focusing principle.

In this method, first devised by V.A. Tsukerman and L.V. Al'tshuler (12), the X rays are focused on a circumference equal to that of the specimen and the X-ray film. Our camera has a conveniently designed stage for taking shots of the specimens by the slide method. The camera can be adapted easily to any existing dismountable X-ray tube by a slight modification of the anticathode cover. There should be a chamfer on the cover so that the primary X-ray beam can be emitted at an angle of 4-8 degrees to the surface of the anode mirror. The camera is fitted with a removable series cassette for six frames. The dimensions of the X-ray film are 13 by 9 centimeters and the diameter of the cassette is 86 millimeters, which corresponds to a threefold scale with respect to the ordinary Debye photographs. The cassette is spring-attached to slide rails mounted securely on the camera, and is easily changed from one frame to another. A movable stage with two windows for taking X-ray photographs by the superimposition method is attached to the upper part of the slide rails.

When taking photographs by the ordinary method, only one of these windows is used, and it remains motionless or oscillates along with the specimen in a plane parallel to its surface. The specimen (slide) can be of any size and does not require special adjusting. It is placed freely on the stage or is held in place by the spring as is the case in the ordinary metal microscope and can be removed easily when changing over for a new photograph without requiring any auxiliary operations.

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An electromagnet, which is secured to the stand separately, can shift the stage of the high-speed camera so that first one and then the other window with the two different specimens comes under the X-ray beam. The time intervals during which the X-ray beam acts on each specimen are accurately regulated by the periodic switching on and off of the electromagnet by means of a contact disc rotated at 6 rpm by a Warren synchronous motor. Provision is made for regulating the time that the magnet is switched on in the range 0.1 to 0.5 of the turning time of the disc. This arrangement allows one to take a series of eleven X-ray photographs by the superimposition method. The time intervals can be varied from 0 to 100 percent in steps of 10 percent. The time taken to switch over from one specimen to the other can be disregarded, as the electromagnet and spring operate quite rapidly. The time intervals are sufficiently small with respect to the overall exposure time, which is equal to several minutes. The electromagnet is fed with direct current from a 0.1-ampere selenium rectifier. The electromagnet and spring can also be used for oscillating the specimen (slide) in which case an alternating current supply is used.

Conclusions

1. New methods and formulas have been suggested which simplify calculations and widen the field of application of the X-ray method of determining the thickness of films.

2. The methods proposed can be extended to cases where the film or the base contains complex chemical compounds whose structure is unknown, solid solutions consisting of light and heavy elements, or when the film or the base is a mixture of several evenly distributed phases.

3. The superimposition method automatically takes into account the states of the crystalline phases of the film and the base which distort the normal values of the intensities of the Debye lines. Such states include disequilibrium of the solid solution, large grain size, dispersion and mosaic of crystalline particles, strain and texture in specimens.

4. A comparative evaluation was made of the accuracy and sensitivity of X-ray methods of determining the thickness of films.

5. Apparatus for taking high-speed X-ray photographs by the superimposition method has been described.

6. The superimposition method has been used to measure the thickness of the Fe_3O_4 layer on oxidized steel.

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